



Serial No. 10/611,934

45360

PATENT

## IN THE UNITED STATES PATENT AND TRADEMARK OFFICE

In re Application of: :  
Sadao KANBE et al. : Art Unit: 1711  
Serial No.: 10/611,934 :  
Filed: July 3, 2003 : Examiner: S. Haider  
For: MICROCAPSULE COMPOSITION FOR ELECTROPHORETIC DISPLAYS

DECLARATION UNDER 37 CFR §1.132

Commissioner for Patents  
P.O. Box 1450  
Alexandria, VA 22313-1450

Sir:

I, Mitsuo KUSHINO, a citizen of Japan, hereby declare and state the following:

1. I am one of co-inventors of U.S. Patent Application Serial No. 10/611,934 filed on July 3, 2003.
2. I am thoroughly familiar with the contents of said Application, its prosecution before the United States Patent and Trademark Office and the references cited therein.
3. I graduated from the Industrial Chemistry course, Wakayama Prefectural Tanabe Technical High School, Wakayama, Japan in the year 1972.
4. I have been employed by Nippon Shokubai Co., Ltd. of Osaka, Japan, one of the assignees of said application in the year 1972 and have been engaged in research and development of microsphere and microparticle at the Research Laboratory of the company.
5. The following experiments were conducted by myself or under my direct supervision and control in order to prove that the microcapsule composition for electrophoretic displays of the present invention exhibits excellent physical properties unexpected from the compositions disclosed in the Hayashi et al. (US 2001/0046081) and Liang et al. (US 2002/0131152).

EXPERIMENTAL:Example 1

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The same procedures as in Example 1 of the present specification were carried out. The physical properties of the microcapsule composition (1) are shown in the following Table I.

#### Example 2

The same procedures as in Example 2 of the present specification were carried out. The physical properties of the microcapsule composition (2) are shown in the following Table I.

#### Example 3

The same procedures as in Example 3 of the present specification were carried out. The physical properties of the microcapsule composition (3) are shown in the following Table I.

#### Example 4

The same procedures as in Example 4 of the present specification were carried out. The physical properties of the microcapsule composition (4) are shown in the following Table I.

#### Comparative Example 1

The same procedures as in Comparative Example 1 of the present specification were carried out. The physical properties of the microcapsule dispersion (c1) are shown in the following Table I.

#### Comparative Example 2

The same procedures as in Comparative Example 2 of the present specification were carried out. The physical properties of the microcapsule dispersion (c2) are shown in the following Table I.

#### Experiment 1

Microcapsules (E1) and a microcapsule dispersion (E1) in which the microcapsules (E1) were dispersed were prepared in the same manner as in Example 1 of the present specification. At this stage, the particle diameters of the microcapsules (E1) were measured in the same manner as in Example 1. As a result, the volume-average particle diameter was 60.1  $\mu\text{m}$ .

The microcapsule dispersion (E1) obtained was concentrated by suction filtration as it was, to give a microcapsule composition (E1) containing the microcapsules (E1) as a filtrated cake. The volume-average particle diameter of the microcapsules (E1) filtrated was 60.2  $\mu\text{m}$ , and the maximum-peak particle diameter was 61.5  $\mu\text{m}$ .

The particle diameter distribution by volume was such that 67 % by volume of the microcapsules were present within the particle diameter range of  $\pm 40$  % of the maximum-peak particle diameter around the maximum-peak particle diameter. The microcapsules

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(E1) were present in a content of 45 % by weight in the microcapsule composition (E1). These results are listed in Table I.

### Experiment 2

Microcapsules (c1) were prepared in the same manner as in Comparative Example 1 of the present specification, and the dried microcapsules (c1) were classified by using a standard mesh having an opening of 75 $\mu$ m and a mesh having an opening of 32 $\mu$ m, to give dried microcapsules (E2).

The volume-average particle diameter of the dried microcapsules (E2) was 56.2 $\mu$ m, and the maximum-peak particle diameter was 57.8 $\mu$ m.

The particle diameter distribution by volume was such that 85 % by volume of the microcapsules were present within the particle diameter range of  $\pm 40$  % of the maximum-peak particle diameter around the maximum-peak particle diameter. These results are listed in Table I.

### Experiment 3

This experiment was reproduced according to paragraphs 0176 to 0178 of Hayashi et al. More specifically, the same procedures as in Example 1 of the present specification were carried out except that dodecylbenzene was changed to Isoper G manufactured by Exxon Chemical Corporation, and anthraquinone blue oil dye was changed to Oil Blue 5502 manufactured by Arimoto Chemical Industry Co., Ltd., to give a dispersion (E3) for electrophoretic displays.

Under stirring with a disperser of Example 1, 36.3 g of the dispersion (E3) for electrophoretic displays was added to 600 g of an aqueous solution which has been previously prepared by dissolving 0.6 g of sodium dodecylbenzene sulfonate as an anionic surfactant and calcium triphosphate as a slightly soluble inorganic salt in water, and the stirring speed was gradually increased to stir the resulting mixture at 2000 rpm for 60 minutes, to give a suspension.

A separable flask was charged with the suspension, and the suspension was heated to 70 °C under nitrogen atmosphere while stirring.

In 104.3 g of an aqueous solution in which potassium persulfate as a polymerization initiator was dissolved 6 g of methyl methacrylate, and the resulting mixture was dispersed with a disperser of Example 1, to give monomer dispersion. The monomer dispersion was added dropwise to the above suspension, the temperature of which was maintained to 70 °C over a period of 20 minutes, and its polymerization reaction was carried out for 7 hours, to give microcapsule dispersion (E3). At this stage, the volume-average particle diameter of the microcapsules (E3) was 110 $\mu$ m.

The microcapsule dispersion (E3) obtained was concentrated by suction filtration as it was, to give a microcapsule composition (E3) containing the microcapsules (E3) as a filtrated paste.

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The volume-average particle diameter of the microcapsules (E3) filtrated was 110.2 $\mu$ m, and the maximum-peak particle diameter was 120.1 $\mu$ m.

The particle diameter distribution by volume was such that 45 % by volume of the microcapsules were present within the particle diameter range of  $\pm 40$  % of the maximum-peak particle diameter around the maximum-peak particle diameter. The microcapsules (E3) were present in a content of 22 % by weight in the microcapsule composition (E3). These results are listed in Table I.

#### **RESULTS:**

As physical properties of the microcapsule composition, ratio (contrast) between reflectances, state of rows of microcapsules, and number of damage or defective microcapsules were determined in the same manner as in the present specification. The results are shown in Table I.

Table I

	Microcapsule Content (wt %)	Volume- average particle diameter ( $\mu\text{m}$ )	Maximum- peak particle diameter ( $\mu\text{m}$ )	Ratio of microcapsules present within particle diameter range of $\pm 40\%$ of maximum-peak particle diameter around it (vol. %)	Physical properties		
					Ratio (contrast) between reflectances	State of rows of microcapsules	Number of damage or defective microcapsules
Ex. 1	45	74.6	77.2	85	5.6	⊙	6
Ex. 2	68	113.2	118.7	81	7.2	⊙	8
Ex. 3	33	70.7	75.5	88	4.7	○	13
Ex. 4	75	121.8	128.1	80	6.9	○	11
Comp. Ex. 1	100	62.1	65.1	51	2.1	×	36
Comp. Ex. 2	100	60.3	61.9	53	2.8	△	28
Experiment 1	45	60.2	61.5	67	2.6	△	11
Experiment 2	100	56.2	57.8	85	2.7	△	36
Experiment 3	22	110.2	120.1	45	1.8	×	28

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**Discussion:**

(1) Experiment 3 is reproduced according to paragraphs 0176 to 0178 of Hayashi et al. This Experiment results in a poor contrast between reflectances, an unacceptable state of rows of microcapsules and an increased number of damaged or defective microcapsules compared to Examples 1 to 4 of the present invention. This fact demonstrates that the present invention exhibits excellent physical properties unexpected from Hayashi et al.

(2) Experiment 1 and Experiment 2 can be compared to Example 1. Example 1 shows that the microcapsule content is 45 % by weight, and that the ratio of microcapsules present within particle diameter range of  $\pm 40$  % of maximum-peak particle diameter is 85 % by volume.

Experiment 1 shows that the microcapsule content, i.e. 45 % by weight is the same as in Example 1, and that the ratio of microcapsules present within particle diameter range of  $\pm 40$  % of maximum-peak particle diameter is different from Example 1.

Experiment 2 shows that the microcapsule content is different from Example 1, and that the ratio of microcapsules present within particle diameter range of  $\pm 40$  % of maximum-peak particle diameter, i.e. 85 % by volume is the same as in Example 1.

Both Experiment 1 and Experiment 2 show poor contrast between reflectances, an unacceptable state of rows of microcapsules and an increased number of damaged or defective microcapsules, as demonstrated in Table I. Therefore, it cannot be expected from these experiments that the contrast between reflectances, the state of rows of microcapsules and the number of damaged or defective microcapsules are improved.

To the contrary, according to Example 1, nevertheless both of 45 % by weight of microcapsule content and 85 % by volume of the ratio of microcapsules present within particle diameter range of  $\pm 40$  % of maximum-peak particle diameter are employed, the contrast between reflectances, the state of rows of microcapsules and the number of damaged or defective microcapsules are highly improved.

This fact proves that the combination of the microcapsule content and the ratio of microcapsules present within particle diameter range of  $\pm 40$  % of maximum-peak particle diameter exhibits excellent physical properties unexpected from Hayashi et al. and Liang et al., because these cited references do not disclose or suggest that the combination exhibits the above excellent physical properties.

Accordingly, the present invention cannot be expected from Hayashi et al. and Liang et al by a person skilled in the art.

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6. The undersigned petitioner declares further that all statements made herein of my own knowledge are true; and that all statements made on information and belief are believed to be true; and further that these statements were made with the knowledge that willful false statements and the like so made are punishable by fine or imprisonment, or both under 1001 of Title 18 of the United States Code and that such willful false statements may jeopardize the validity of the application or any patent issued thereon.

7. Further declare saith not.

  
Mitsuo KUSHINO

12. Mar. 2008  
Date